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(54) **CELLULOSE FIBRES AND FILAMENTS HAVING A HIGH ELONGATION AT BREAK**

CELLULOSEFASERN UND FILAMENTEN MIT HOHER BRUCHDEHNUNG

FIBRES ET FILAMENTS DE CELLULOSE A COEFFICIENT ELEVE D'ALLONGEMENT A LA
RUPTURE

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Description

[0001] The invention pertains to cellulose fibres obtainable by spinning a solution containing the constituents cellulose, a solvent, and water.

[0002] JP-A-07189019 discloses the production of cellulose fibres from a spinning solution prepared by dissolving cellulose in an N-methylmorpholine-N-oxide-water mixed solvent. Though the properties of the fibres obtained with the known process display satisfactory results, there is a constant need for further improvement.

The invention now provides cellulose fibres and yarns displaying improved mechanical properties.

The invention consists in that cellulose fibres of the type mentioned in the opening paragraph, which are obtained from a spinning solution containing 94-100 wt.% of the constituents cellulose, phosphoric acid and/or its anhydrides, and water, have an elongation at break of more than 7%.

[0003] It should be added that non-prepublished patent application WO 96/06208 discloses cellulose fibres, filaments, and yarns obtained by spinning an anisotropic cellulose solution obtainable by dissolving cellulose in a solvent containing 65-80 wt.% of phosphorus pentoxide. Said fibres, filaments, and yarns are characterised by a comparatively high breaking tenacity (> 500 mN/tex), a comparatively high modulus (> 15 N/tex), and a comparatively low elongation at break ($< 7\%$). A drawback of fibres, filaments, and yarns displaying said properties is that they are less suitable for use in textiles due to, int. al., the relative discomfort attending the wearing of such high-modulus fibres, and particular technical applications due to their comparatively low elongation at break.

[0004] The term phosphoric acid in this patent application refers to all inorganic acids of phosphorus and their mixtures. Orthophosphoric acid is the acid of pentavalent phosphorus, i.e. H_3PO_4 . Its anhydrous equivalent, i.e. the anhydride, is phosphorus pentoxide (P_2O_5). In addition to orthophosphoric acid and phosphorus pentoxide there is, depending on the quantity of water in the system, a series of acids of pentavalent phosphorus with a water-binding capacity in between those of phosphorus pentoxide and orthophosphoric acid, such as polyphosphoric acid ($H_6P_4O_{13}$, PPA).

[0005] The weight percentage of phosphorus pentoxide in the solvent is calculated by starting from the overall quantity by weight of phosphoric acid including its anhydrides and the total quantity of water in the solvent, converting the acids into phosphorus pentoxide and water, and calculating the percentage of said overall quantity by weight made up by phosphorus pentoxide.

In this description water derived from cellulose or from substances which are part of the other constituents and water which is added to obtain the solution are not included in the calculation of the concentration of phosphorus pentoxide in the solvent.

[0006] The weight percentage of phosphorus pentoxide in the solution is calculated by starting from the overall quantity by weight of phosphoric acid including its anhydrides and the total quantity of water in the solution, converting the acids into phosphorus pentoxide and water, and calculating which percentage of said overall quantity by weight is made up by phosphorus pentoxide. For that reason in this description water derived from cellulose or from substances which are part of the other constituents and water which is added to obtain the solution are included in the calculation of the concentration of phosphorus pentoxide in the solution.

[0007] The weight percentage of cellulose in the solution is calculated by starting from the overall quantity by weight of all constituents in the solution.

[0008] Cellulose derivatised with phosphoric acid is included among the constituents making up 94-100 wt.% of the solution to be spun.

In the case of cellulose derivatised with phosphoric acid the percentages by weight of cellulose in the solution listed in this patent specification refer to quantities calculated back on the cellulose. This applies in analogous fashion to the quantities of phosphorus mentioned in this specification.

[0009] In addition to water, phosphoric acid and/or its anhydrides, and cellulose and/or reaction products of phosphoric acid and cellulose, other substances may be present in the solution.

The solution can be prepared by mixing constituents classifiable into four groups: cellulose, water, phosphoric acid including its anhydrides, and other constituents. The "other constituents" may be substances which benefit the processability of the cellulose solution, solvents other than phosphoric acid, or adjuvants (additives), e.g., to counter cellulose degradation as much as possible, or dyes and the like.

[0010] Preferably, the solution is composed of 96-100 wt.% of the constituents cellulose, phosphoric acid and/or its anhydrides, and water.

Preferably, no solvents other than phosphoric acid are employed, and adjuvants or additives are present only in amounts of 0 to 4 wt.%, calculated on the overall quantity by weight of the solution. More favoured still is a solution containing the lowest possible quantity of substances other than the constituents cellulose, phosphoric acid and/or its anhydrides, and water, i.e., with from 0 to 1 wt.% of additives.

[0011] It was found that cellulose fibres according to claim 1 can be obtained by spinning isotropic as well as anisotropic cellulose solutions, i.e. solutions on basis of cellulose.

These isotropic and anisotropic solutions can be obtained extremely simply and within a short period of time making use of cellulose and a solvent containing 65-85 wt.% of phosphorus pentoxide, with the phosphorus pentoxide content in the solvent being calculated in relation to the overall quantity of phosphorus pentoxide and water in the solvent. In obtaining cellulose fibres according to claim 1 there was found to be a difference between isotropic and anisotropic solutions.

Isotropic solutions

[0012] Isotropic, spinnable cellulose solutions containing 94-100 wt.% of the constituents cellulose, phosphoric acid and/or its anhydrides, and water can be spun using a so-called dry jet-wet spinning process. In such a spinning process the following steps can be distinguished:

- the solution is extruded through one or more capillaries,
- the extrudate is passed through a layer containing a non-coagulating medium (e.g., a layer of air), in which layer the extrudate can be drawn,
- the extrudate is coagulated in a coagulating liquid (to form fibres and/or endless filaments),
- the fibres/filaments thus formed are washed and gathered/wound.

[0013] It was found that when a spinnable, isotropic cellulose solution is employed in such a spinning process, the following have particular significance when it comes to obtaining fibres/filaments having an elongation at break of more than 7%:

- The extrudate is not drawn in the layer containing a non-coagulating medium or drawn only to a very limited extent. Preferably, the extrudate is relaxed somewhat in this layer.
- The coagulating liquid contains less than 50 wt.% of water, preferably less than 10 wt.% of water. In a very favourable preparative process the coagulating liquid will be anhydrous (i.e., the liquid will contain less than 5 wt.% of water), or the coagulating liquid is water with cations added thereto, preferably an aqueous solution which contains monovalent cations as, e.g., Li^+ , Na^+ or K^+ . Such solutions can be obtained, e.g., by solving lithium, sodium, or potassium phosphate in water.
- The fibres/filaments are washed and wound under the lowest possible tension, preference being given to tension-free washing and/or winding of the fibres/ filaments.

[0014] Suitable for use as coagulants containing less than 50 wt.% water for extrudates obtained from an isotropic, spinnable solution are low-boiling, a-polar organic liquids which have only a limited swelling effect on cellulose or mixtures thereof. Examples of such suitable coagulants include alcohols, ketones, esters or mixtures thereof. The use of acetone as coagulant is preferred. Optionally, water may be added to the coagulant in order to obtain a coagulant containing less than 50 wt.% of water. However, it is preferred to employ a coagulant containing less than 10 wt.% of water, more particularly, a coagulant which is essentially anhydrous.

Suitable for use as coagulants containing water and cations added thereto for extrudates obtained from an isotropic, spinnable solution are aqueous solutions containing monovalent cations as, e.g., Li^+ , Na^+ or K^+ . Such solutions can be obtained, e.g., by solving lithium, sodium, or potassium phosphate in water.

Anisotropic solutions

[0015] Anisotropic cellulose solutions containing 94-100 wt.% of the constituents cellulose, phosphoric acid and/or its anhydrides, and water can also be spun using a dry jet-wet spinning process.

It was found that when an anisotropic cellulose solution is employed in such a spinning process, the following have particular significance when it comes to obtaining fibres/filaments having an elongation at break of more than 7%:

- The coagulating liquid contains mostly water optionally with cations added thereto, preferably contains monovalent cations added thereto, or the coagulating liquid contains less than 50 wt.% of water, preferably less than 10 wt.% of water.
- The fibres/filaments are washed and wound under the lowest possible tension.
- If the coagulating liquid contains less than 50 wt.% of water, the fibres/filaments are coagulated under low tension.

[0016] Suitable for use as coagulants containing water and cations added thereto for extrudates obtained from an anisotropic, spinnable solution are aqueous solutions containing monovalent cations as, e.g., Li^+ , Na^+ or K^+ . Such solutions can be obtained, e.g., by solving lithium, sodium, or potassium phosphate in water.

Suitable for use as coagulants containing less than 50 wt.% water for extrudates obtained from an anisotropic, spinnable solution are low-boiling, a-polar organic liquids which have only a limited swelling effect on cellulose or mixtures thereof. Examples of such suitable coagulants include alcohols, ketones, esters or mixtures thereof. The use of acetone as coagulant is preferred. Optionally, water may be added to the coagulant in order to obtain a coagulant containing less than 50 wt.% of water. However, it is preferred to employ a coagulant containing less than 10 wt.% of water, more particularly, a coagulant which is essentially anhydrous.

It was found that the in the spinning of anisotropic solution the use of a coagulant containing less than 50 wt.% of water, more particularly, a coagulant which is essentially anhydrous, enables the preparation of cellulose fibres and filaments with an elongation at break above 7% and a breaking tenacity above 600 mN/tex, more in particular a breaking tenacity above 700 mN/tex.

[0017] A single spinneret plate having the desired number of capillaries may be used not only for extruding cellulose fibres and filaments having an elongation at break of more than 7% from isotropic as well as anisotropic solutions, but also for extruding the cellulose multifilament yarns much in demand in actual practice which have an elongation at break of more than 7% and contain from 30 to 10 000, preferably from 100 to 2000, filaments. The manufacture of such multifilament yarns preferably is carried out on a cluster spinning assembly containing a number of spinning orifice clusters, as described in EP 168 876 or on a spinning assembly with one or more spinnerets, which spinnerets are described in WO 95/20969.

[0018] Following coagulation the formed fibres/filaments may be washed. While the washing liquids may be selected from the same group of low-boiling organic solvents or mixtures of these solvents as used as coagulants, the preferred washing liquid is water.

[0019] After the solution has been coagulated and washed, the resulting product may be neutralised, e.g., by washing it with a solution of $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ in water.

[0020] The resulting fibres, filaments, and yarns have particularly favourable properties, especially for textile applications and particular technical applications. In addition to an elongation at break of more than 7% these products have a breaking toughness of more than 10 J/g, more particularly a breaking toughness of more than 15 J/g.

It was found that because of the composition of the spinning solution the fibres contain at least 0,02 wt.% of cellulose-bound phosphorus. Also, the fibres generally have a low modulus and because of the presence of cellulose-bound phosphorus exhibit good flame retardance, good dye uptake, and good moisture absorption. The fibres, filaments, and yarns exhibit substantially better flame retardance than cotton or well-known synthetic cellulose fibres such as viscose yarn. The flame retardance of the fibres, filaments or yarns can be measured, e.g., by means of an LOI test.

Measuring methods

Determination of isotropy/anisotropy

[0021] Visual determination of the isotropy or anisotropy was performed with the aid of a polarisation microscope (Leitz Orthoplan-Pol (100x)). To this end about 100 mg of the solution to be defined were arranged between two slides and placed on a Mettler FP 82 hot-stage plate, after which the heating was switched on and the specimen heated at a rate of about 5°C/min. In the transition from anisotropic to isotropic, i.e., from coloured (birefringent) to black, the temperature is read off at virtual black. The transition temperature is indicated as T_{ni} .

The visual assessment during the phase transition was compared with an intensity measurement using a photosensitive cell mounted on the microscope. For this intensity measurement a specimen of 10-30 μm was arranged on a slide such that no colours were visible when crossed polarisers were employed. Heating was carried out as described above. The photosensitive cell, connected to a recorder, was used to write the intensity as a function of time. Above a certain temperature (differing for the different solutions) there was a linear decrease of the intensity. Extrapolation of this line to an intensity of 0 gave the T_{ni} . In all cases, the value found proved a good match for the value found by the above-mentioned method.

Isotropic solutions do not display birefringence at room temperature. This means that T_{ni} will be below 25°C. However, it may be the case that such solutions do not display an isotropy/anisotropy transition.

Determination of DP

[0022] The degree of polymerisation (DP) of the cellulose was determined with the aid of an Ubbelohde type 1 ($k=0,01$). To this end the cellulose specimens to be measured were dried *in vacuo* for 16 hours at 50°C after neutralisation, or the amount of water in the copper II ethylene diamine/water mixture was corrected to take into account the water in the cellulose. In this way an 0,3 wt.% of cellulose-containing solution was made using a copper II ethylene diamine/water mixture (1/1). On the resulting solution the viscosity ratio (visc. rat. or η_{rel}) was determined, and from this the limiting viscosity (η) was determined in accordance with the formula:

$$[\eta] = \frac{\text{visc.rat} - 1}{c + (k \times c \times (\text{visc.rat} - 1))} \times 100$$

wherein

c = cellulose concentration of the solution (g/dl) and

k = constant = 0,25

[0023] From this formula the degree of polymerisation DP was determined as follows:

$$DP = \frac{[\eta]}{0,42} \text{ (for } [\eta] < 450 \text{ ml / g),}$$

or

$$DP^{0,76} = \frac{[\eta]}{2,29} \text{ (for } [\eta] > 450 \text{ ml / g)}$$

[0024] Determining the DP of the cellulose in the solution proceeded as described above after the following treatment: 20 g of the solution were charged to a Waring Blender (1 litre), 400 ml of water were added, and the whole was then mixed at the highest setting for 10 minutes. The resulting mixture was transferred to a sieve and washed thoroughly with water. Finally, there was neutralisation with a 2%-NaHCO₃ solution for several minutes and after-washing with water to a pH of about 7. The DP of the resulting product was determined as described above, starting from the preparation of the copper II ethylene diamine/water/cellulose solution.

Determination of phosphorus content

[0025] The quantity of phosphorus bound to the cellulose in the solution, or in a cellulose product made using said solution, can be determined by 300 mg of cellulose solution, which solution has been coagulated and, after thorough washing for 16 hours at 50°C. dried *in vacuo* and then stored in a sealed sampling vessel, being combined in a decomposition flask with 5 ml of concentrated sulphuric acid and 0,5 ml of an Yttrium solution containing 1000 mg/l of Yttrium. The cellulose is carbonised with heating. After carbonisation hydrogen peroxide is added to the mixture in portions of 2 ml, until a clear solution is obtained. After cooling the solution is made up with water to a volume of 50 ml. ICP-ES (Inductive Coupled Plasma - Emission Spectrometry) is used to measure, by means of a phosphorus calibration line determined using reference samples containing 100, 40, 20, and 0 mg/l of phosphorus, respectively, the phosphorus content in the solution to be measured with the aid of the following equation:

$$\text{phosphorus content (\%)} = (P_{\text{conc}}(\text{mg/l}) \times 50) / (C_w(\text{mg}) \times 10)$$

wherein:

P_{conc} = the phosphorus concentration in the solution to be measured and

C_w = the weighed out quantity of coagulated and washed cellulose.

[0026] Yttrium is added as internal standard to correct the solutions' viscosity variations. The phosphorus content is measured at a wavelength of 213,6 nm, the internal standard is measured at a wavelength of 224,6 nm.

Mechanical properties

[0027] The mechanical properties of the filaments and the yarns were determined in accordance with ASTM standard D2256-90, using the following settings.

The filament properties were measured on filaments clamped with Arnitel® gripping surfaces of 10 × 10 mm. The filaments were conditioned for 16 hours at 20°C and 65% relative humidity. The length between grips was 100 mm, the filaments were elongated at a constant elongation of 10 mm/min.

The yarn properties were determined on yarns clamped with Instron 4C clamps. The yarns were conditioned for 16 hours at 20°C and 65% relative humidity. The length between grips was 500 mm, the yarns were elongated at a constant elongation of 50 mm/min. The yarns were twisted, the number of twists per meter being 4000/linear density [dtex].

The linear density of the filaments, expressed in dtex, was calculated on the basis of the functional resonant frequency (ASTM D 1577-66, Part 25, 1968); the yarn's linear density was determined by weighing.

The tenacity, elongation, and initial modulus were derived from the load-elongation curve and the measured filament or yarn linear density.

The initial modulus (In. Mod.) was defined as the maximum modulus at an elongation of less than 2%. The final modulus was defined as the maximum modulus at an elongation of more than 2%.

Examples

[0028] The invention will be illustrated with reference to examples.

[0029] Unless otherwise specified, the following starting materials were employed to prepare the solutions in the examples.

Material	Manufacturer and product code	Content P ₂ O ₅ [%]
P ₂ O ₅	J.T. Baker, 0193	98
H ₃ PO ₄	La Fonte Electrique SA, Bex Suisse crystallised, >99% (98,3% analysed)	71,2
H ₄ P ₂ O ₇	Fluka Chemika, 83210, 97% (98,8% anal.)	78,8
PPA*	Caldic	84,5

*PPA = polyphosphoric acid

Example 1

[0030] An isotropic cellulose solution obtained by the process described in the non-published patent application NL 1002236 in the name of Applicant, containing 7,6 wt.% of cellulose (Alphacell C-100, DP=2300), was extruded at 40°C through a spinneret having 375 capillaries each with a diameter of 65 µm. The extruded solution was passed through an air gap and coagulated in a bath filled with acetone of about 35°C. After coagulation the multifilament yarn formed was washed with water, neutralised with a 2,5 wt.% Na₂CO₃·10H₂O solution in water, and washed again with water. The yarn was then dried under very low tension.

Several experiments were carried out with different draw ratios in the air gap, the draw ratio being defined as the throughput rate in the coagulation bath divided by the rate at which the solution was extruded from the capillaries. The mechanical properties of the resulting yarns were measured. The data is listed in Table 1.

Table 1

	DR	LD [dtex]	BT [mN/tex]	EaB [%]	IM [N/tex]	FM [N/tex]	Bto [J/g]
1a	0,6	3320	180	17,4	3,5	1,6	18,6
1b	0,9	2390	270	11,5	5,7	3,0	17,0
1c	1,1	1770	310	9,5	7,6	4,2	16,1
1d	1,5	1370	380	8,4	9,2	6,0	16,6

wherein DR = draw ratio, LD = Linear density, BT = Breaking tenacity, EaB = Elongation at break, IM = Initial modulus, FM = Final modulus, and Bto = Breaking toughness.

Example 2

[0031] An anisotropic cellulose solution obtained by the process described in non-published patent application WO 96/06208 in the name of Applicant, containing 18 wt.% of cellulose (Buckeye V60, DP=820), was extruded at 55°C through a spinneret having 250 capillaries each with a diameter of 65 µm. The extruded solution was passed through an air gap and coagulated in a coagulation bath in a solution of 2 wt.% of Na₃PO₄ in water of 12°C. The extrudate was drawn 5,7x in the air gap. The resulting yarn was washed with water, finished, and dried at 150°C. During washing and drying the tension exerted on the yarn was kept as low as possible. The yarns from Examples 2e and 2g were dried tension-free.

During the experiment the degree of acidity of the coagulating liquid increased. The mechanical properties of the resulting yarns were measured. The data is listed in Table 2.

Table 2

	pH	LD [dtex]	BT [mN/tex]	EaB [%]	IM [N/tex]	FM [N/tex]	Bto [J/g]
2a	11,5	495	300	7,5	14,8	3,5	15,1
2b	9,4	490	300	7,5	15,0	3,5	15,1
2c	7,3	485	300	7,6	15,0	3,6	15,3
2d	6,7	485	295	7,4	14,7	3,6	14,8
2e	5,9	510	265	8,6	8,5	3,0	13,6
2f	5,5	515	260	7,3	13,0	3,1	12,9
2g	2,8	515	250	8,3	8,3	2,9	12,6

wherein pH = degree of acidity of the coagulating liquid, LD = Linear density, BT = Breaking tenacity, EaB = Elongation at break, IM = Initial modulus, FM = Final modulus, and Bto = Breaking toughness.

Example 3

[0032] An anisotropic cellulose solution containing 18 wt.% of cellulose (Buckeye V60, DP=820), obtained by the process described in non-prepublished patent application WO 96/06208 in the name of Applicant, i.e., by mixing cellulose with a solvent containing 73,9 wt.% P_2O_5 , was extruded at 46°C through a spinneret having 375 capillaries each with a diameter of 65 μ m. The extruded solution was passed through an air gap and coagulated in a coagulation bath. The resulting yarn was washed with water, finished, and dried at 150°C. During washing and drying the tension exerted on the yarn was kept as low as possible.

During the experiment the composition of the coagulant in the coagulation bath was changed. Some yarns were neutralised with a 2,5 wt.% $Na_2CO_3 \cdot 10H_2O$ solution in water.

The mechanical properties of the resulting yarns were measured. The data is listed in Table 3.

Table 3.

	Coagulation bath water +	T _{coag} [°C]	neutr.	BT [mN/tex]	EaB [%]	IM [N/tex]	Bto [J/g]
3a	-	18	-	276	9,2	13,0	16,9
3b	-	18	+	280	9,6	13,0	17,8
3c	5 gew.% $ZnSO_4$	23	-	260	10.1	12.0	
3d	5 gew.% $ZnSO_4$	23	+	260	10.6	11.5	

wherein T_{coag} = temperature of the coagulating liquid, BT = Breaking tenacity, EaB = Elongation at break, IM = Initial modulus, and Bto = Breaking toughness.

Example 4

[0033] An anisotropic cellulose solution containing 18 wt.% of cellulose (Buckeye V60, DP=820), obtained by the process described in non-prepublished patent application WO 96/06208 in the name of Applicant, i.e., by mixing cellulose with a solvent containing 74,2 wt.% P_2O_5 , was extruded through a spinning assembly with four spinnerets, each spinneret having 375 capillaries each with a diameter of 65 μ m. The extruded solution was passed through an air gap and coagulated in acetone. The resulting yarns were washed with water, neutralised by treatment with an Na_2CO_3 solution, washed again with water some yarns were finished, all yarns were dried, and wound onto a bobbin with a speed of 100 m/min

The spinning conditions are given in Table 4A.

Table 4A,

spinning conditions				
Example	T _{coag} [°C]	neutralisation solution	finish step	drying step
4a	13,5	2,5 wt.% Na_2CO_3	yes, RT32A	tensionless, 25°C
4b	12,5	0,5 wt.% Na_2CO_3	no	160°C on heated godet
4c	-22,1	2,5 wt.% Na_2CO_3	yes, RT32A	160°C on heated godet

The mechanical properties of the resulting yarns were measured. The data is listed in Table 4B.

Table 4B,

mechanical properties					
Example	LD [dtex]	BT [mN/tex]	EaB [%]	IM [N/tex]	Bto [J/g]
4a	2510	750	7,5	15,1	27,0
4b	2570	882	7,4	18,5	31,6
4c	2653	846	7,1	21,4	30,5

wherein LD = Linear density, BT = Breaking tenacity, EaB = Elongation at break, IM = Initial modulus, and Bto = Breaking toughness.

Claims

1. A cellulose fibre obtained by spinning a solution containing the constituents cellulose, a solvent, and water, characterised in that the fibre obtained from a spinning solution containing 94-100 wt.% of the constituents cellulose, phosphoric acid and/or its anhydrides, and water has an elongation at break of more than 7%.
2. A cellulose fibre according to claim 1, characterised in that the fibre is an endless filament.
3. A cellulose fibre according to claim 1 or 2, characterised in that the breaking toughness of the fibre exceeds 10 J/g.
4. A cellulose yarn obtained by spinning a solution containing the constituents cellulose, a solvent, and water, characterised in that the yarn obtained from a spinning solution containing 94-100 wt.% of the constituents cellulose, phosphoric acid and/or its anhydrides, and water has an elongation at break of more than 7%.
5. A cellulose yarn according to claim 4, characterised in that the yarn contains more than 50 filaments.
6. A cellulose yarn according to claim 4 or 5, characterised in that the breaking toughness of the yarn exceeds 10 J/g.
7. A cellulose yarn according to claim 6, characterised in that the breaking toughness of the yarn exceeds 15 J/g.
8. A process for preparing cellulose fibres or filaments by spinning a solution on the basis of cellulose, characterised in that the solution contains 94-100 wt.% of the constituents cellulose, phosphoric acid and/or its anhydrides, and water, and wherein the solution is coagulated in a liquid which contains water with cations added thereto.
9. A process according to claim 8, characterised in that an isotropic solution on the basis of cellulose is used.
10. A process according to claim 8 or 9, characterised in that the solution is spun using a dry jet-wet spinning process.
11. A process according to any one of claims 8-10, characterised in that the solution is coagulated in water with mono-valent cations added thereto.
12. A process according to any one of claims 8-11, characterised in that after coagulation the fibres or filaments are washed and dried under low tension.
13. A process according to claim 12, characterised in that the fibres or filaments are washed in water.

Patentansprüche

1. Cellulosefaser, erhalten durch Verspinnen einer Lösung, welche die Komponenten Cellulose, ein Lösungsmittel und Wasser enthält, dadurch gekennzeichnet, dass die aus einer Spinnlösung, die 94 bis 100 Gew.% der Komponenten Cellulose, Phosphorsäure und/oder deren Anhydride und Wasser enthält, erhaltene Faser eine Bruchdehnung von mehr als 7% besitzt.

2. Cellulosefaser nach Anspruch 1, dadurch gekennzeichnet, dass die Faser ein Endlosfilament ist.
3. Cellulosefaser nach Anspruch 1 oder 2, dadurch gekennzeichnet, dass die Reissfestigkeit der Faser grösser als 10 J/g ist.
4. Cellulosegarn, erhalten durch Verspinnen einer Lösung, welche die Cellulosekomponenten, ein Lösungsmittel und Wasser enthält, dadurch gekennzeichnet, dass das aus einer Spinnlösung enthaltend 94 bis 100 Gew.% der Komponenten Cellulose, Phosphorsäure und/oder deren Anhydride und Wasser erhaltene Garn eine Bruchdehnung von mehr als 7% aufweist.
5. Cellulosegarn nach Anspruch 4, dadurch gekennzeichnet, dass das Garn mehr als 50 Filamente enthält.
6. Cellulosegarn nach Anspruch 4 oder 5, dadurch gekennzeichnet, dass die Reissfestigkeit des Garnes grösser als 10 J/g ist.
7. Cellulosegarn nach Anspruch 6, dadurch gekennzeichnet, dass die Reissfestigkeit des Garnes grösser als 15 J/g ist.
8. Verfahren zur Herstellung von Cellulosefasern oder Filamenten durch Verspinnen einer Lösung auf Basis von Cellulose, dadurch gekennzeichnet, dass die Lösung 94 bis 100 Gew.% der Komponenten Cellulose, Phosphorsäure und/oder deren Anhydride und Wasser enthält, wobei die Lösung in einer Flüssigkeit koaguliert wird, die Wasser mit zugesetzten Kationen enthält.
9. Verfahren nach Anspruch 8, dadurch gekennzeichnet, dass eine isotrope Lösung auf Basis von Cellulose verwendet wird.
10. Verfahren nach Anspruch 8 oder 9, dadurch gekennzeichnet, dass die Lösung unter Anwendung eines Trockendüsen-Nassspinnverfahrens versponnen wird.
11. Verfahren nach einem der Ansprüche 8 bis 10, dadurch gekennzeichnet, dass die Lösung in Wasser mit diesem zugesetzten einwertigen Kationen koaguliert wird.
12. Verfahren nach einem der Ansprüche 8 bis 11, dadurch gekennzeichnet, dass die Fasern oder Filamente nach der Koagulation unter geringer Spannung gewaschen und getrocknet werden.
13. Verfahren nach Anspruch 12, dadurch gekennzeichnet, dass die Fasern oder Filamente in Wasser gewaschen werden.

Revendications

1. Une fibre de cellulose obtenue par filage d'une solution contenant comme constituants de la cellulose, un solvant et de l'eau, caractérisée en ce que la fibre obtenue à partir d'une solution de filage contenant 94-100% de cellulose, acide phosphorique et/ou ses anhydrides et de l'eau comme constituants, présente un allongement à la rupture supérieur à 7%.
2. Une fibre de cellulose selon la revendication 1, caractérisée en ce que la fibre est un filament continu.
3. Une fibre de cellulose selon la revendication 1 ou 2, caractérisée en ce que la ténacité à la rupture de la fibre dépasse 10 J/g.
4. Un fil de cellulose obtenu par filage d'une solution contenant, comme constituants de la cellulose, un solvant et de l'eau, caractérisée en ce que le fil obtenu à partir d'une solution de filage contenant 94-100% en poids de cellulose, d'acide phosphorique et/ou ses anhydrides et de l'eau comme constituants, a un allongement à la rupture supérieur à 7%.
5. Un fil de cellulose selon la revendication 4, caractérisé en ce que le fil comporte plus de 50 filaments.

6. Un fil de cellulose selon une quelconque des revendications 4 ou 5, caractérisé en ce que la ténacité à la rupture du fil dépasse 10 J/g.
7. Un fil de cellulose selon la revendication 6, caractérisé en ce que la ténacité à la rupture du fil dépasse 15 J/g.
8. Un procédé de préparation de fibres ou de filaments de cellulose par filage d'une solution à base de cellulose, caractérisé en ce que la solution contient 94-100% en poids de cellulose, acide phosphorique et/ou ses anhydrides, et eau comme constituants, et dans lequel la solution est coagulée dans un liquide qui contient de l'eau additionnée de cations.
9. Un procédé selon la revendication 8, caractérisé en ce qu'on utilise une solution isotrope à base de cellulose.
10. Un procédé selon la revendication 8 ou 9, caractérisé en ce que la solution est filée en utilisant un procédé à jet sec-filage humide.
11. Un procédé selon une quelconque des revendications 8 à 10, caractérisé en ce que la solution est coagulée dans l'eau additionnée de cations monovalents.
12. Un procédé selon une quelconque des revendications 8 à 11, caractérisé en ce que, après coagulation, on lave les fibres ou filaments et on les sèche sous faible tension.
13. Un procédé selon la revendication 12, caractérisé en ce qu'on lave les fibres ou filaments dans l'eau.